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FAST-SETTING POURABLE MORTARS WITH HIGH FLUIDITY**FIELD OF THE INVENTION**

The present invention relates to the field of cementitious compositions. Described herein are compositions suitable for forming cementitious mortars having a high

5 degree of fluidity and a rapid development of resistance.

PRIOR ART

Pourable mortars are liquid compositions characterized by a high degree of fluidity. They are used in the cement sector for specific applications in which a very fluid mix is required, which is able to reach crevices and narrow spaces to fill

10 them in a homogeneous way and solidify therein. Examples of said applications are the recovery of deteriorated building works, consolidation of rock formations, structural reinforcement, injection in the conduits of tendons, immobilization of toxic-noxious refuse (e.g., asbestos), and the production of cementitious products for pouring in moulds.

15 Pourable mortars in general consist of hydraulic binders, aggregates having a diameter not greater than 4 mm, water, and possibly, added substances and additives. Amongst the additives there may be listed: fluidifiers, superfluidifiers, setting correctors, substances that facilitate adhesion to the substrate, air-entraining agents, expansive agents, etc.

20 Examples of pourable cementitious mortars known in the state of the art are *Mapegrout colabile* (pourable Mapegrout) (manufactured by Mapei S.p.A.) and *Malta antiritiro reoplastica autolivellante* (self-levelling rheoplastic anti-shrink mortar) (manufactured by Siriobeton A.C); Macflow Rheomac 200 (manufactured by MAC S.p.A.) is marketed as specific binder for making pourable mortars.

25 The performance of the mortars in the fresh state is evaluated using testing methods for measuring spreading by means of a vibrating table (UNI 7044-72) or for determining consistency by means of a funnel groove (UNI 8997). Both of the methods provide an indication of the consistency through the measurement of a dimension characteristic of the area occupied by a pre-set volume of mortar on a

30 horizontal plane and in defined conditions of testing.

The high degree of fluidity of pourable mortars, albeit desirable for applications referred to above, can also present some disadvantages; for example, in the case of consolidation of vertical or inclined surfaces, the mortar, once applied, tends to be dispersed from the site of application before hardening. In the case of the moulding of products, a great deal of time is required before the fluid mass solidifies and develops resistance such as to enable extraction of the product and disengagement of the mould. It would thus be useful to accelerate hardening of the mortar, for example by means of the addition of setting accelerators; however, said operation tends to reduce the fluidity of the mortar.

10 There is therefore the need to increase the speed of consolidation of pourable mortars, without this having the repercussion of an undesired reduction in fluidity of the product, and at the same time safeguarding the mechanical properties of the hardened product.

15 Another need is to obtain extremely fluid mortars, without resorting to large amounts of fluidifiers/superfluidifiers. The use of said additives in large quantities considerably increases the cost of the cementitious mix and can lead to modifications in the performance of the consolidated product.

SUMMARY

20 The present Applicant has surprisingly found that by mixing with water a fast-setting cement, fluidifiers and/or superfluidifiers, setting regulators, and aggregates having a specific granulometric distribution, mortars are obtained characterized by a high degree of fluidity and short consolidation times.

25 The aggregates used are made up of two fractions with different grain size: specifically, the ratio between the characteristic grain diameters of the two fractions is comprised between 2.2 and 3.2 (said ratio is calculated by setting in the numerator the granulometric fraction with the larger diameter).

30 Each of the two fractions is preferably substantially monogranular, i.e., the particles that compose it have a negligible variation in diameter with respect to the characteristic value of the fraction. The mortars thus obtained have values of fluidity 2-3 times higher than those of mortars produced with the granulometric

distribution of traditional aggregates. Said increases in fluidity are obtained without modifying the water/cement ratio or the amount of fluidifiers used.

The mortars produced according to the invention may be used to advantage in all applications in which it is required to associate a high degree of fluidity to a rapid consolidation. A preferred example of said applications is the production of cementitious products by means of pouring in moulds. The high degree of fluidity enables homogeneous filling of the moulds even without any need for vibration for compacting the mass. The high degree of fluidity does not interfere with the time of setting of the cement: consequently, after pouring, the composition hardens and develops the necessary resistance over the short time, thus enabling a rapid disengagement of the moulds and a faster production cycle.

DESCRIPTION OF FIGURES

Figure 1: granulometric distribution of the traditional aggregates used as comparison in Example 1

Figure 2: fractions of aggregate in accordance with the invention, used in Example 2

DETAILED DESCRIPTION OF THE INVENTION

A first subject of the present invention is a dry premix, useful for forming pourable mortars, comprising a fast-setting hydraulic binder, fluidifiers and/or superfluidifiers, setting regulators, and aggregates.

From the mineralogical standpoint, the aggregates are the ones commonly used in the preparation of concretes (e.g., sand) and are classified in the standard UNI 8520.

Said aggregates are not used as such in a coarse form, but are previously divided on the basis of the grain size. For the formation of the compositions in question two fractions of aggregates having specific grain sizes are used. Of determining importance is the fact that the ratio between the characteristic grain diameters of the two fractions of aggregates should be comprised between 2.2 and 3.2, preferably between 2.5 and 3.0.

For the purposes of the present invention, by the term "characteristic grain diameter" for a given fraction of aggregates (also defined herein as X_0) is meant

the opening of mesh [expressed in mm] of the screen at which the cumulative undersize (P_c) for that given fraction is equal to 63.2%.

As regards the absolute value in millimetres of the characteristic grain diameter, useful and non-limiting ranges of reference are: between 0.2 mm and 0.4 mm for

5 one fraction, and between 0.6 mm and 0.8 mm for the other fraction; however, any fraction having a characteristic grain diameter of up to 4 mm approximately may be used in the invention, provided that the dimensional ratio between the characteristic grain diameters of the two fractions is comprised between 2.2, and 3.2.

10 It has moreover been observed that the effectiveness of the invention increases, the more the fractions tend to be homogeneous in diameter, i.e., tending towards monogranularity. It is thus preferable that, for each fraction, there should be a small variation in diameter with respect to the characteristic value. Monogranularity is conveniently expressed by the parameter n . The parameter n 15 is obtained via the RRSB equation¹ (DIN 66145):

$$P_c = 100 \left[1 - e^{-\left(\frac{x}{x_0}\right)^n} \right]$$

where:

P_c = cumulative undersize [%];

x = opening of mesh of the screen [expressed in mm];

20 x_0 = characteristic grain diameter [expressed in mm], as defined previously;

n = form parameter of the granulometric distribution.

According to the present invention the equation RRSB is used, applying the least-squares algorithm (see Comincioli, Analisi Numerica - Metodi Modelli Applicazioni - McGraw-Hill - 1990, pp. 166-167), to interpolate the granulometric distribution

25 determined experimentally and deducing as interpolation parameters the values of x_0 and n . The resulting values of x_0 and n , for a given distribution, are assumed as

¹ RRSB: initials of Rosin, Ramler, Sperling and Bennet

distinctive of said distribution. In particular, the parameter n is an index of the quantity of particles that, in a given granulometric distribution, are characterized by values of diameters different from x_0 . In particular, as n increases, the number of particles having a diameter other than x_0 decreases and, consequently, the 5 distribution tends to be monogranular, with a diameter tending to x_0 . In particular, it is preferable that the parameter n should be greater than or equal to 4.0: said value indicates fractions that, for the purposes of the present invention, are considered substantially monogranular. It is moreover preferable that the two 10 fractions be equally represented, i.e., that approximately one half of the total aggregates (meaning by "approximately one half" 40-60 wt% with respect to the total of the aggregates) be made up of the first fraction and the remaining half approximately (i.e., the corresponding 60-40 wt%) should consist of the second fraction.

With respect to the weight of the total dry premix, the aggregates are incorporated 15 in a weight percentage of between 45 wt% and 65 wt%, preferably between 50 wt% and 54 wt%.

In the compositions according to the invention, any fast-setting hydraulic binder may be used. By "hydraulic binder" is meant a pulverized cementitious material in the solid, dry state, which, when mixed with water, yields plastic mixes capable of 20 promoting setting and hardening. Fast-setting hydraulic binders are products in themselves known and widely used in the cement sector. The Italian Ministerial Decree dated August 31, 1972 ("Norme sui requisiti di accettazione e modalità di 25 prova degli agglomerati cementizi e delle calci idrauliche" - *Rules on the requisites of acceptance and modalities of testing of cementitious agglomerates and hydraulic limes*) defines the requisites of said binders, namely, that they must possess start-setting times longer than 1 minute, and end-setting times shorter than 30 minutes, determined on normal paste, and that they must moreover have a minimum resistance to compression at 7 days of at least 13 MPa; further 30 specifications regard the content of SO_3 (less than 3.5 wt%) and of MgO (less than 4 wt%). Fast binders are characterized in general by high contents of calcium aluminate; there are moreover known fluoroaluminate-based fast binders

(Italian patent No. IT 37815 A/69 and Italian patent No. IT 988018). A preferred example of fast-setting binder is the lime-and-aluminate-based composition described in the patent No. EP-A-1159233 (incorporated herein for reference) and referred to herein as "Scatto". The fast-setting binder may be used as such or

5 possibly mixed with ordinary cement (defined according to the standard UNI EN 197-1, for example Portland cement (CEM I)), photocatalytic cements, possible further reactive added substances (e.g., anhydrite, silica smoke, added substances of a pozzolan nature, of Type II, as defined by the standard UNI EN 206.)

10 The fast-setting binder is incorporated in the compositions of the invention in weight percentages with respect to the total dry mix comprised between 15 wt% and 30 wt%, preferably between 20 wt% and 24 wt%.

The binder/aggregates weight ratio, herein meaning by "binder" the sum of fast-setting binder plus the possible ordinary or photocatalytic cement present and the 15 possible further added substances described above is generally comprised between 0.65 and 1.00, preferably between 0.90 and 0.95.

15 The fluidifiers/superfluidifiers are used in weight percentages comprised between 0.2 wt% and 4 wt% with respect to the total weight of the binder. Examples of these additives are the compounds of a melaminic, naphthalenic, or acrylic type 20 commonly used in cementitious compositions. They may be used individually or mixed with two or more of them. The fluidifiers/superfluidifiers may be indifferently added to the starting dry premix or may be added mixed with the water at the moment of preparation of the mortar.

The setting regulators are used in weight percentages comprised between 25 0.01 wt% and 0.4 wt% with respect to the total weight of the binder.

By way of non-limiting example, among the setting regulators there may be listed: citric acid, boric acid and tartaric acid.

In addition to the aforesaid components, the premix forming the subject of the present invention may contain various additives to enable fine adaptation of the 30 characteristics of the cement to the specific application required. Examples of said additives are: waterproofing agents, organic resins, air-entraining agents,

expansive agents, etc. Said products are useful but not indispensable for the purposes of the invention.

The compositions identified above are mixed with water, obtaining low-viscosity and fast-hardening cementitious mortars. Said mortars form a further subject of 5 the present invention.

The ratio of mixing with water can vary widely: non-limiting ranges of reference are comprised between 0.30 and 0.45, preferably between 0.34 and 0.38. By "ratio of mixing with water" or "water/binder ratio" is meant the ratio, respectively, between the quantity of water used for forming the mortar (including the water 10 possibly contributed through the addition of aqueous additives) and the quantity of "binder" present, as defined previously, where the amount of water is the numerator and the amount of binder the denominator.

It is important to note that the mortars according to the present invention reach a high fluidity, without requiring the use of large amounts of water; consequently, it 15 is possible to obtain hardened end products with an excellent resistance thanks to the low water/binder ratio used.

The mortars according to the invention may be produced through any process that envisages mixing of their components: processes and apparatus commonly in use for the formation of cementitious mortars may be used. The temperature at which 20 mixing of the dry premix with water occurs is generally comprised between 5°C and the 35°C.

Useful applications for the mortars according to the invention are the ones envisaged for known pourable mortars, where the present invention affords the advantage of an improved fluidity and of a faster development of resistance. 25 Examples of such uses are the recovery of deteriorated building works, consolidation of rock formations, structural reinforcement, injection in the conduits of tendons, immobilization of toxic-noxious refuse (e.g., asbestos), etc., and the production of cementitious products for pouring in moulds. In the latter case, the high degree of fluidity of the mortar enables the fluid mass to reach 30 homogeneously all the interstices that the shape envisages, thus producing products that are precise in shape, even in the case of complex and tortuous

shapes and ones having a particularly high degree of surface finish; at the same time, rapid consolidation enables a faster disengagement of the moulds and hence a faster production cycle.

Other useful applications in which the high speed of setting of the mortar is
5 exploited are, for example, the laying of manhole covers, the fixing of brackets, sanitary piping and hinges, the setting in place and/or fixing of wooden and metal frames, the setting in place of junction boxes and conduits for electrical installations, the sealing of cement ducts or piping, drains or cisterns, the blocking of infiltrations of water, the coating of road surfaces or tracks, the covering of
10 roofs, and the preparation of products made of concrete.

The present invention also includes new cementitious products, obtained starting from the new cementitious compositions described above. Said products are characterized in that they contain a fast-setting binder and the two fractions of aggregates as defined above, and possess excellent mechanical properties. The
15 invention is now described by means of the following non-limiting examples.

EXPERIMENTAL PART

Characteristics of known pourable mortars

Reference is made to the following widely used pourable mortars:

- MC1. mortar with a base of MACFLOW (RHEOMAC 200) – MAC S.p.A.;
- 20 MC2. MAPEGROUT COLABILE – MAPEI S.p.A.;
- MC3. MALTA ANTIRITIRO REOPLASTICA AUTOLIVELLANTE (SELF-
LEVELLING RHEOPLASTIC ANTI-SHRINK MORTAR) – SIRIOBETON
A.C.

The mortars referred to above are specifically designed for providing high
25 mechanical resistance, together with characteristics of pourability. Table 1 below summarizes the performance declared in the technical sheets of the products. In particular, for the binder MC1 the performance for two different compositions is given:

- MC1-A) performance declared in paste (mix consisting of binder and water
30 characterized by a water/binder ratio = 0.32);

MC1-B) performance found in mortar (mix consisting of binder, aggregates proportioned according to Fuller distribution and water; binder/aggregate ratio = 1:1.25, water/binder ratio = 0.38).

Table 1: Characteristics of known mortars

Product code	Mechanical performance			Rheological characteristics
	Expiry [days]	R _{compression} [MPa]	R _{flexure} [MPa]	
MC1-A (declared)	1	20	-	Enables very fluid and non-segregatable concretes with a low water/cement ratio to be obtained.
	28	65	-	
MC1-B (found)	0.25	not measurable	not measurable	Flow time (Marsh cone): 94 s
	1	31.4	6.2	
	7	58.9	8.2	
MC2	1	35.0	5.5	High degree of fluidity, suitable for application by means of pouring in formwork, without segregation, even in large thicknesses.
	7	60.0	8.0	
MC3	28	75.0	10.0	Pourable mortar for the renewal of concrete.
	7	-	≥ 4	
	28	≥ 65	-	

Example 1 (reference)

In order to verify the criticality of the granulometric curve of the aggregates made up of two fractions and of the dimensional ratio between the two fractions (such as is the subject of the present invention) reference compositions were obtained 10 which contained a different number of fractions of aggregates.

Specifically, three reference compositions (referred to as "Mix 15", "Mix 16", and "Mix 26") were prepared containing a fast-setting binder ("Scatto"), an ordinary Portland cement, and some additives used in cementitious compositions, such as anhydrite, silica-smoke slurry, and superfluidifiers; the aggregates used in the

5 three compositions were characterized by the granulometric distributions given in Figure 1 and described hereinafter:

Mix 15: granulometric distribution obtained by forming three different fractions of aggregates, characterized by the parameters x_0 and n given in Table 2 below. The same table gives, for each fraction, the weight percentage content referred to the

10 mix of aggregate alone.

Table 2 : Granulometric fractions used for Mix 15

	Fractions of aggregate		
	C	B	D
x_0 [mm]	0.23	0.73	1.33
n	1.7	5.0	8.2
r^2	> 0.99	> 0.99	> 0.99
content in wt% of aggregate alone in the mix	67	20	13

For each fraction of aggregate, Table 2 also gives the value of r^2 (determination index). Said value, which is always comprised between 0 and 1, is an index of the

5 goodness of an interpolation of discrete data performed by means of a given function. In the specific case, it refers to the interpolation of the experimental data of grain size, performed using the RRSB equation; its value, which is close to 1, indicates the excellent agreement between said equation and the granulometrical curves considered.

10 **Mix 16:** granulometric distribution obtained by forming three different fractions of aggregates, characterized by the parameters x_0 and n given in Table 3 below. The same table gives, for each fraction, the weight percentage content referred to the mix of aggregate alone.

Table 3: Granulometric fractions used for Mix 16

	Fractions of aggregate		
	A	E	D
x_0 [mm]	0.27	0.97	1.33
n	4.7	3.5	8.2
r^2	> 0.99	> 0.99	> 0.99
content in wt% of aggregate alone in the mix	66	29	5

Mix 26: granulometric distribution obtained by forming four different fractions of aggregates, characterized by the parameters x_0 and n given in Table 4 below. The same table gives, for each fraction, the weight percentage content referred to the mix of aggregate alone.

5 **Table 4: Granulometric fractions used for Mix 26**

	Fractions of aggregate			
	A	B	F	D
x_0 [mm]	0.27	0.73	2.00	1.33
n	4.7	5.0	4.7	8.2
r^2	> 0.99	> 0.99	> 0.99	> 0.99
Content in wt% of aggregate alone in the mix	50	20	20	10

The compositions Mix 15, Mix 16 and Mix 26 were mixed with water so as to obtain mortars with water/binder ratios in the region of 0.33-0.36. For each mortar, there were measured, in the fresh state, the volumic mass and the flow time, the 10 latter factor being indicative of the fluidity and measured according to what is specified in what follows.

The mortar, immediately after its preparation, was poured (approximately 1.1 litres) into a metal cone in compliance with the standard UNI EN 445 (Marsh cone). There was then found the flow time of a known and pre-set volume 15 (nominally, 1 litre) of mortar through the nozzle set in a position corresponding to the vertex of the cone.

The mortars were then poured to form prisms having dimensions of 40 x 40 x 160 mm and in conformance with the standard UNI EN 196-1 (without settling). The mortar was kept in the moulds located in a climatized environment 20 ($T = 20 \pm 2^\circ\text{C}$; $\text{RH} = 50 \pm 5\%$) up to removal from the moulds after 2 h 45 min from casting. The specimens were then kept in the same climatized environment up to

expiry of the 24 hours. For the conduct of resistance-to-compression tests for longer expiration times, the specimens were set to cure in water at $20\pm2^\circ\text{C}$.

On the specimens there was measured the development of resistance to compression at 3, 6 and 24 hours. The results are set forth in Table 5 below.

5 **Table 5: Compositions and parameters of the reference mortars**

	Mix 15	Mix 16	Mix 26
Scatto [%]	15.85	15.85	19.60
CEM I 52.5 R [%]	17.10	17.10	20.00
Anhydrite [%]	1.30	1.30	1.70
Silica-smoke slurry (dry substance) [%]	0.85	0.85	0.85
Citric acid⁽¹⁾ [%]	0.20	0.20	0.25
Aggregate (see Tables 2,3,4) [%]	51.70	51.70	43.10
Water [%]	12.60	12.60	14.05
Acrylic superfluidifier (dry substance) [%]	0.40	0.40	0.45
Water/binder ratio	0.36	0.36	0.33
Volumic mass [kg/m³]	2210	2220	2220
Flow time [s]	180	113	145
Resistance to compress. [MPa]: 3 h	3.3	3.1	2.8
Resistance to compress. [MPa]: 6 h	13.1	12.7	12.9
Resistance to compress. [MPa]: 24 h	20.1	19.5	25.0

(1) : added in 50% aqueous solution

The compositions of Table 5 were defined so as to minimize the viscosity of the mixes, and consequently the flow times, and at the same time limit any possible

10 phenomena of segregation. In any case, it may be noted that the flow time (113-180 s) was decidedly unsatisfactory.

Example 2

A mortar was prepared in accordance with the present invention (referred to herein as "MBV"), using amounts of ingredients equivalent to the ones used in the reference examples; however, in this case an aggregate was used, obtained by 5 mixing just two distinct fractions of aggregate, designated in Figure 2 by the letters A and B. The ratio between the characteristic grain diameters of the two fractions is equal to 2.70.

Table 6 gives the granulometric distribution of the two fractions A and B.

Table 6: Granulometric distribution of the mortar MBV

Diameter (mm)	Fraction A	Fraction B
1.6	100.0	100.0
1.0	100.0	99.3
0.5	100.0	13.5
0.25	49.4	1.4
0.2	21.1	1.1
0.125	3.1	0.7
0.075	0.8	0.4
0.051	0.0	0.0

10

The fraction A represents 49 wt% and the fraction B represents 51 wt% with respect to the total aggregates.

Table 7 gives the characteristic parameters of the curve of granulometric distribution illustrated in Figure 2. The same table gives, for each fraction, the 15 weight percentage content referred to the mix of aggregate alone.

Table 7: Granulometric fractions used for the mortar MBV

	Fraction	
	A	B
x_0	0.27	0.73

<i>n</i>	4.7	5.0
<i>r</i> ²	> 0.99	> 0.99
Content in wt% of aggregate alone in the mix	49	51

From Table 7 it may be noted that the two distributions are characterized by a value of the parameter $n > 4.0$, which confirms the substantial monogranularity of the distributions. The characteristic grain diameters for the two distributions are 5 respectively equal to 0.27 mm and 0.73 mm. The ratio between the characteristic grain diameters of the two distributions is equal to 2.70.

The composition data, the values of volumic mass, of fluidity of the mortar (expressed in terms of flow time through the Marsh cone), and the values of resistance of the product in the hardened state are set forth in Table 8. The 10 determination of these parameters was performed as in Example 1.

Table 8: Composition and parameters of the mortar MBV

MBV		
Scatto	[%]	18.60
CEM I 52.5 R	[%]	19.81
Anhydrite	[%]	1.49
Silica-smoke slurry (dry substance)	[%]	0.90
Citric acid ⁽¹⁾	[%]	0.12
Aggregate	[%]	44.20
Water	[%]	14.40
Acrylic superfluidifier (dry substance)	[%]	0.48
a/l		0.35
Volumic mass	[kg/m ³]	2214
Flow time	[s]	58
Resistance to compression	[MPa]	3 h 11.4

8 h	15.6
24 h	19.0
7 days	65.6
28 days	77.3

(1): added in 50% aqueous solution

The flow time (58 s, indicating a high degree of fluidity) was completely different from and lower than those measured for the reference compositions (113-180 s).

5 There was thus obtained a fluidity twice or three times that of the reference compositions illustrated in Table 5. Apart from the different grain size of the aggregates, the various compositions tested were substantially equivalent from the standpoints of quality and quantity. In particular, the water/cement ratio used was kept in the region of 0.33-0.36, and the amounts of superfluidifiers were kept

10 in the region of 0.4-0.5 wt%. The marked increases in fluidity of the mortar were thus a consequence of the modifications made in the grain size of the aggregates. It may moreover be noted that said substantial increase in fluidity was not matched with any reduction in the development of resistance which, with respect to the reference compositions, remained unvaried at 24 hours and even improved

15 over the short time. The development of resistance over the short time is of primary importance for the purpose of solving the problems addressed by the present invention, widely discussed in the foregoing detailed description.

The values of fluidity observed for the mortar MBV are compatible with its use for forming homogeneous products in moulds, without any application of vibrations

20 for settling the mass. In particular, the resistance observed at 3 hours (> 10 MPa) is sufficient to enable extraction of the solidified product from the mould and its manipulation.

When subjected to freezing/thawing tests (according to UNI 7087), the products presented, after 300 cycles, a factor of degradation approaching 1.0, which

25 indicates a good resistance to freezing. The shrinkage data of the samples stored at 20°C and 50% relative humidity yielded values equivalent to standard mortars.

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